



Short communication

The fabrication of hierarchical NiMoO₄@Ni(OH)₂ nanocomposites and its electrochemical behavior used as supercapacitor electrode

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ARTICLE INFO

Article history:

Received 10 October 2017

Received in revised form 21 November 2017

Accepted 22 November 2017

Available online 24 November 2017

Keywords:

Nickel molybdate

Nickel hydroxide

Electrochemical impedance spectroscopy

Energy storage and conversion

ABSTRACT

A hierarchical nanocomposites of NiMoO₄@Ni(OH)₂ is fabricated via hydrothermal and electrodeposition method. The electrochemical tests performed using cyclic voltammetry, chronopotentiometry and electrochemical impedance spectroscopy display that the hierarchical NiMoO₄@Ni(OH)₂ nanocomposites exhibits superior supercapacitive performance to those of NiMoO₄ and Ni(OH)₂, respectively. The initial specific capacitance for NiMoO₄@Ni(OH)₂ is 1278 F g⁻¹ at a current density of 1 mA cm⁻². The Nyquist plot of the NiMoO₄@Ni(OH)₂ reveals low R_{ct} and its EIS data fitted by Zsimpwin software matched well with the primary data.

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Supercapacitors, also known as electrochemical capacitors (ECs), have attracted much attention in recent years due to their high power density, high energy density and long cycle stability [1,2]. On account of the crucial impact of the electrodes on the electrochemical properties of supercapacitors, many efforts have been made to explore various potential electrode materials, such as carbon-based and transitional metal-based materials [3]. Nevertheless, the former exhibits low capacitance and low energy densities due to the narrow cell voltage range so that the investigation has been focused on the latter [4–6]. However, due to the intrinsic poor electrical conductivity of transitional metal oxides and the short diffusion distance of electrolyte into electrodes, lots of active materials cannot be involved in the effective faradaic reactions.

Recently, metal molybdates have been incrementally studied as promising electrode owing to their low cost, abundant resources and environmental friendly features [7–9]. Among them, NiMoO₄ reveals high specific capacitance due to its high electrochemical activity [8]. However, the rate capability and cycle performance of NiMoO₄ are not satisfactory so that lots of attempt have been devoted to improve its electrochemical property. To construct nanocomposites is an effective method. It is well-known that the hybridization of NiMoO₄ with other capacitive materials is attractive because the formation of composite with versatile and tunable properties is superior to the individual material. Furthermore, this is an effective approach to utilize the advantages of the respective electrodes. Ni(OH)₂ is another promising electrode with high theoretical specific capacitance and superior stability in strong alkaline electrolyte. Moreover, Ni(OH)₂ is often hybridized with

other materials due to its high reversibility and excellent rate capability when oxidized to NiOOH [10,11].

Here in this work, hierarchical NiMoO₄@Ni(OH)₂ nanofibers are successfully fabricated via hydrothermal and electrodeposition method. Electrochemical tests indicate that the NiMoO₄@Ni(OH)₂ nanofibers, with low charge-transfer resistance, exhibits high capacitance and excellent cycling stability as an electrode material for supercapacitors in 2 M KOH electrolyte, which makes it a promising candidate for the fabrication of electrochemical energy-storage devices.

The XRD patterns of NiMoO₄ and NiMoO₄@Ni(OH)₂ are shown in Fig. 1a. It can be clearly seen that the three strongest diffraction peaks at 44.4°, 51.8° and 76.4° are ascribed to the (111), (200) and (220) crystal planes of metallic nickel, respectively [12]. Obviously, the XRD signals corresponding to the active materials on the Ni foam substrates are affected by the high intensity of the above mentioned peaks. Even so, the characteristic signal peaks related to the active materials can also be found. Among them, the peaks located at 27.3°, 29.8° and 61.6° are attributed to NiMoO₄ (JCPDS Card No.13–0128). Nevertheless, the crystallinity is not perfect and inevitably leads to more crystal deficiencies, which may be beneficial to the transportation of electrolyte ions into the internal space of the active materials and form larger pseudocapacitance area [13]. The peaks at 19.2°, 38.6°, 59.0° and 66.8° are referred to (001), (101), (110) and (111) crystal planes of Ni(OH)₂ (JCPDS Card No.14–0117). The two types of diffraction peaks corresponding to NiMoO₄ and Ni(OH)₂ reveal the existence of NiMoO₄@Ni(OH)₂.

The typical SEM images of NiMoO₄ samples are shown in Fig. 1b, which presents irregular straw-like nanofibers. After electrodeposition of Ni(OH)₂ onto the NiMoO₄, the morphology of the obtained NiMoO₄@Ni(OH)₂ are similar to that of the pure NiMoO₄, which are

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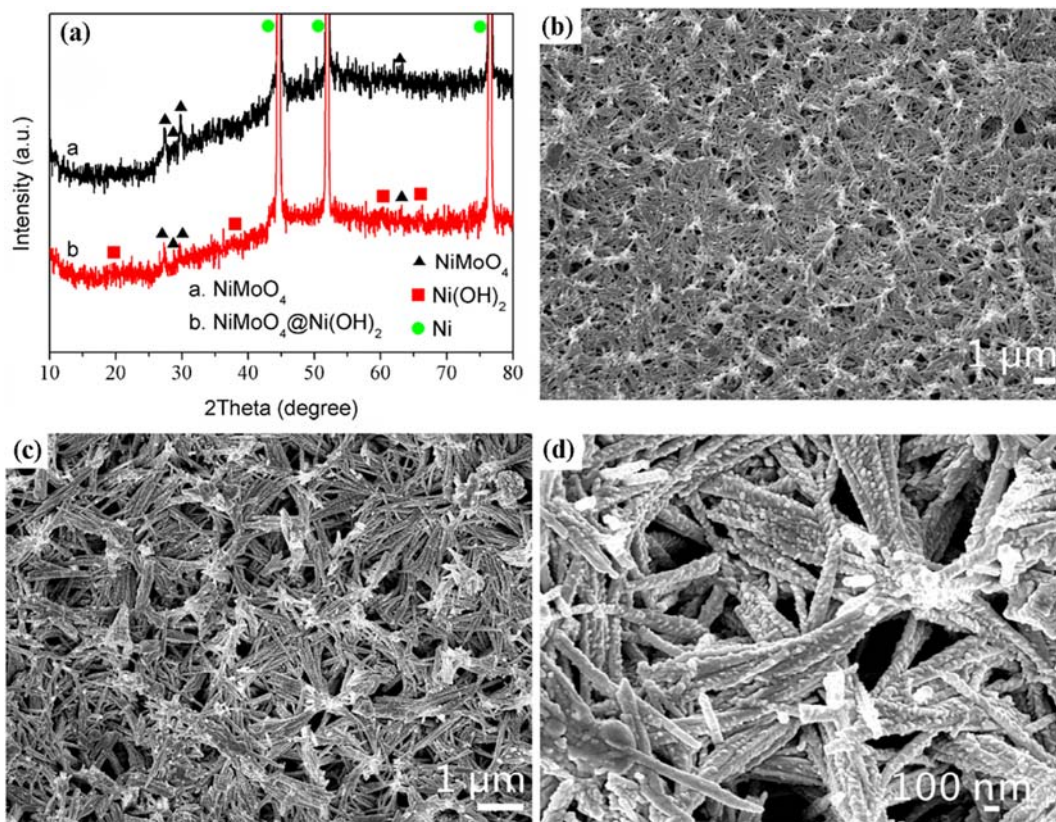


Fig. 1. (a) XRD patterns of NiMoO₄ and NiMoO₄@Ni(OH)₂, (b) and (c) are SEM images of NiMoO₄ and NiMoO₄@Ni(OH)₂, (d) is the enlarged area form (c).

displayed in Fig. 1c and d. Nevertheless, the surface of the NiMoO₄@Ni(OH)₂ sample is coarse with plenty of small uniform nanoparticles (shown in Fig. 1d), which may enhance the specific surface area and the contact surface between the electrode material and the electrolyte, so that the capacitance may also be further improved.

In order to investigate the electrochemical performance of the NiMoO₄@Ni(OH)₂ nanocomposite, the CV tests of NiMoO₄, Ni(OH)₂ and NiMoO₄@Ni(OH)₂ electrodes are carried out at a scan rate of 5 mV s⁻¹, as shown in Fig. 2a. It is obvious that the CV curve area for the NiMoO₄@Ni(OH)₂ is bigger than that of the NiMoO₄ and Ni(OH)₂ electrodes, indicating the superior electrochemical performances of the composite electrode. The specific capacitances of the three electrodes calculated according to the galvanostatic charge-discharge curves at a current density of 1 mA cm⁻² is shown in Fig. 2b. It is clear that NiMoO₄@Ni(OH)₂ possesses the highest specific capacitance of 1250 F g⁻¹ which is greatly higher than that of NiMoO₄ and Ni(OH)₂ at the same current density. EIS measurements are performed to evaluate the electrochemical performance of NiMoO₄@Ni(OH)₂, NiMoO₄ and Ni(OH)₂. The Nyquist plots of the three electrodes are displayed in Fig. 2c. Normally, the Nyquist plots are composed of a semicircle in the high-frequency range and a straight line in the low-frequency range [14]. Charge transfer resistance (R_{ct}), related to faradic reactions, can be got from the diameter of semicircle. In this study, the semicircle corresponding to NiMoO₄@Ni(OH)₂ and NiMoO₄ are not evident, which is similar to the reported result [15], indicating the lower R_{ct} than that of the Ni(OH)₂. The intersection of the semicircle on the real axis represents the equivalent series resistance (R_s) of the electrochemical system, which includes the ionic resistance of the electrolyte, the intrinsic resistance of the active material and the contact resistance at the interface between active material and current collector [7]. The straight line at the low-frequency represents the diffusion resistance (Warburg impedance, R_w). The slope of the straight line on the NiMoO₄@Ni(OH)₂ Nyquist plot is greater than the other two electrodes, meaning the fast

ion diffusion from electrolyte solution into the interior of the electrode, which implies the ideally capacitive behavior of the NiMoO₄@Ni(OH)₂ nanocomposite electrode.

CV measurement at different scan rates are carried out to further investigate the electrochemical properties of the NiMoO₄@Ni(OH)₂ nanocomposite. As shown in Fig. 2d, two strong redox reaction peaks can be seen in the CV curves at low scan rates, indicating its pseudocapacitive performance. The anodic peak distorts as the increase of scan rate, which is due to the reason that the peak value of the electricity and voltage are both separate at high scan rates, on account of the polarization and ohmic resistance in the course of faradic reactions [16]. For the sake of further explore the impedance behavior of the NiMoO₄@Ni(OH)₂ nanocomposite, the EIS data for NiMoO₄@Ni(OH)₂ is fitted by Zsimpwin software and the matched circuit is demonstrated in Fig. 3a. The parameters of R_s , R_{ct} , R_w and CPE are the equivalent series resistance, charge-transfer resistance, Warburg resistance and the constant phase angle element, respectively [17]. In fact, the equivalent circuit is generally more complex employing non-physical circuit elements such as Warburg impedances and constant phase elements [18]. Nevertheless, it can provide an intuitional circuit diagram to help us to understand the possible electrode electrochemical process. The cycling performance of NiMoO₄@Ni(OH)₂ is displayed in Fig. 3b at a current density of 2 mA cm⁻². During the first 300 cycles, the capacitance decay severely and thereafter the capacitance variation tend to steady. The capacitance loss may be ascribed to the following reasons: (1) the faradic reactions during the charge/discharge process is irreversible; (2) the structure of the active materials collapses during the cycling process [19,20].

In conclusion, a hierarchical NiMoO₄@Ni(OH)₂ nanocomposites has been prepared by hydrothermal and electrodeposition method. The NiMoO₄@Ni(OH)₂ nanocomposites electrode exhibits excellent electrochemical performances to NiMoO₄ and Ni(OH)₂ electrodes. The cyclic voltammetry and galvanostatic charge-discharge tests display the

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